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IS 1448-52 (1971): Methods of test for petroleum and its products, Part 52: Drop Point [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 52]

DROP POINT

(First Revision)

Third Reprint NOVEMBER 1991
(Incorporating Amendment No. 1)

UDC 665·6/·7 : 536·421·3

Adapted from the Joint Publication ASTM Designation D 566/64 and IP Designation 132/65

[This revision is being issued in order to align the method with the joint ASTM—IP Standard]

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1. SCOPE

1.1 This method is intended for the determination of drop point of lubricating greases. It should not be considered as having any bearing upon service performance.

2. TERMINOLOGY

2.1 For the purpose of this standard, the drop point shall be the temperature at which the grease passes from a semi-solid to a liquid state under the conditions of the test.

3. OUTLINE OF METHOD

3.1 The sample, contained in the grease cup, is heated until a drop falls from the orifice of the cup.

4. APPARATUS

4.1 The apparatus shall consist of the following parts assembled as shown in Fig. 1.

4.1.1 *Grease Cup* — a chromium plated brass cup conforming to the dimensions as shown in Fig. 2.

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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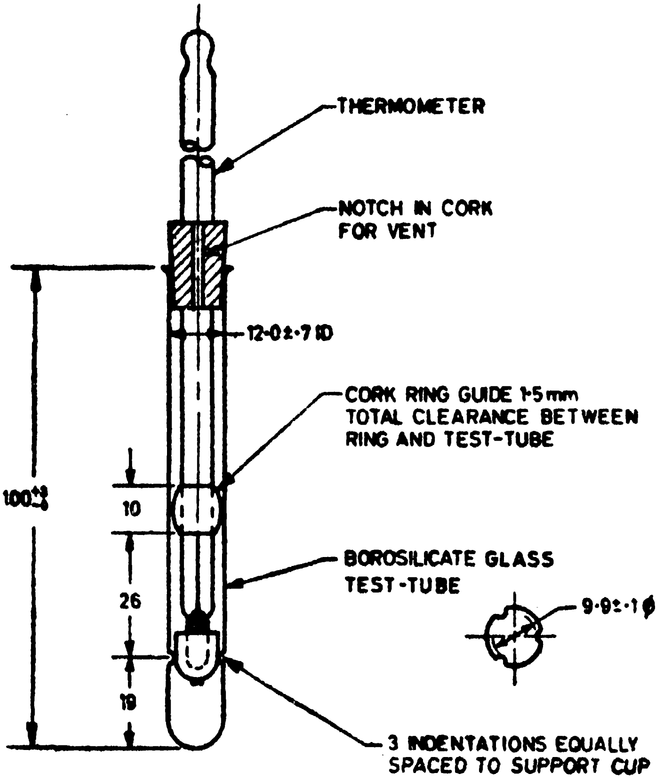
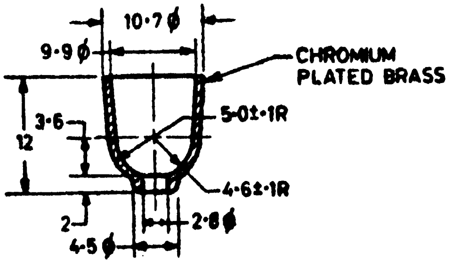

THERMOMETER
NOTCH IN CORK FOR VENT
12·0±·7 ID
CORK RING GUIDE 1·5 mm TOTAL CLEARANCE BETWEEN RING AND TEST-TUBE
100+3 -0
10
26
19
BOROSILICATE GLASS TEST-TUBE
9·9±·1 ϕ
3 INDENTATIONS EQUALLY SPACED TO SUPPORT CUP

All dimensions in millimetres.

FIG. 1 ASSEMBLY OF APPARATUS



All dimensions in millimetres.

FIG. 2 DIMENSION OF GREASE CUP

4.1.2 *Test-Tube* — of heat resistant glass with rim and with its mouth fitted with a cork having a notch as shown in Fig. 1. The tube shall be 100 to 103 mm in length and 12·0 ± 0·7 mm in inside diameter having three indentations about 19 mm from the bottom, equally spaced on the circumference. The depth of these indentations shall be such as to support the grease cup in the test-tube. The test-tube shall also have a cork ring guide as shown in Fig. 1.

4.1.3 *Thermometers* — two, conforming to the following requirements:

Range	−5°C to +300°C
Graduation	1°C
Immersion	76 mm
Overall length	305 to 395 mm
Stem diameter	6·0 to 7·5 mm
Bulb shape	Cylindrical
Bulb length	10 to 15 mm
Bulb diameter	5·0 to 6·0 mm
Length of graduated portion	222 to 262 mm
Distance, bottom of bulb to 0°C	100 to 110 mm
Longer lines at each	5°C
Figured at each	10°C
Expansion chamber	Required
Top finish	Ring
Scale error not to exceed	± 1°C

4.1.4 *Rod* — of polished metal, 150 mm long, 1·2 to 1·6 mm in diameter.

4.1.5 *Bath* — an oil-bath consisting of a 400-ml beaker and suitable oil as the heating medium and provided with means for heating and stirring the liquid. Heating is preferably done by an immersed electrical resistance heater regulated by voltage control.

5. PROCEDURE

5.1 Place the corks on one of the thermometers as shown in Fig. 1 and adjust the position of the upper cork so that the tip of the thermometer bulb will be about 3 mm above the bottom of the grease cup when the apparatus is assembled for the test. Suspend a second thermometer in the oil-bath so that its bulb will be at approximately the same level as the bulb of the thermometer in the test-tube.

Note — The position of the tip of the thermometer in the test-tube is not critical so long as the orifice is not obstructed; with the film of grease applied to the inside of the cup, the thermometer bulb should not be in contact with the grease.

5.2 Remove the grease cup and fill by pressing the larger opening into the grease to be tested until the cup is filled, taking care to avoid working of the grease as far as possible. Remove excess grease with a spatula or knife. Gently press the cup, held in a vertical position with the smaller opening at the bottom, down over the metal rod until the latter protrudes about 25 mm. Press the rod against the cup in such a manner that the rod makes contact at both the upper and lower peripheries of the cup. Maintain this contact, rotating the cup on the rod along the index finger so as to give it a spiral-like motion down the rod to remove a conical section of the grease which adheres along the rod. When the cup has finally slipped over the end of the rod, a smooth film of reproducible thickness shall be left inside the cup.

5.3 Place the cup and the thermometer in the test-tube and suspend the test-tube in the oil-bath with the oil level within 6 mm of the rim. If the cork holding the thermometer in the test-tube has been properly chosen, the 76 mm immersion mark on the thermometer will coincide with the lower edge of the cork. The assembly should be immersed to this point.

5.4 Stir the oil-bath and heat at a rate of 4 to 7°C per minute until the bath reaches a temperature approximately 20°C below the expected dropping point of the grease. At this point, reduce the rate of heating so that the temperature in the test-tube will be within 2°C or less of the temperature in the oil-bath before the oil-bath temperature increases an additional 3°C. Continue heating at a rate such that the difference between the temperatures in the test-tube and in the oil-bath is maintained between 1 and 2°C. This condition is established when the oil-bath is heated at a rate of about 1·0 to 1·5°C per minute. As the temperature increases, material will gradually protrude through the orifice of the grease cup. When a drop of material falls, note the temperatures on the two thermometers and record their average as the dropping point of the grease.

NOTE 1 — Certain greases, for example, some aluminium-base greases, form a drop with a tailing thread upon melting, which may break off or which may hold until the drop reaches the bottom of the test-tube; in any case, the dropping point shall be taken as the temperature when the drop reaches the bottom of the test-tube.

NOTE 2 — The dropping points of some greases, particularly those containing aluminium soaps, are known to decrease upon ageing, the change being much greater than the deviation permitted in results obtained by different laboratories. Therefore, comparative tests between laboratories should be made within a period of six days.

5.5 Two determinations may be made simultaneously in the same bath, provided both samples have approximately the same dropping points.

6. CALCULATION AND REPORTING

6.1 Calculate and report the mean of all the determinations to nearest 0·5°C as the drop point.

7. PRECISION

7.1 Results of duplicate tests shall not differ by more than the following amounts:

Drop Point Range, °C	*Deviation*, °C	
	Repeatability	Reproducibility
88 to 105	2	5
106 to 200	7	12
201 and above	7	20

Reprography Unit, BIS, New Delhi, India